



U.S. Army Research, Development and Engineering Command



# ***TECHNOLOGY DRIVEN. WARFIGHTER FOCUSED.***

## Nanotechnology Capabilities at Picatinny Arsenal

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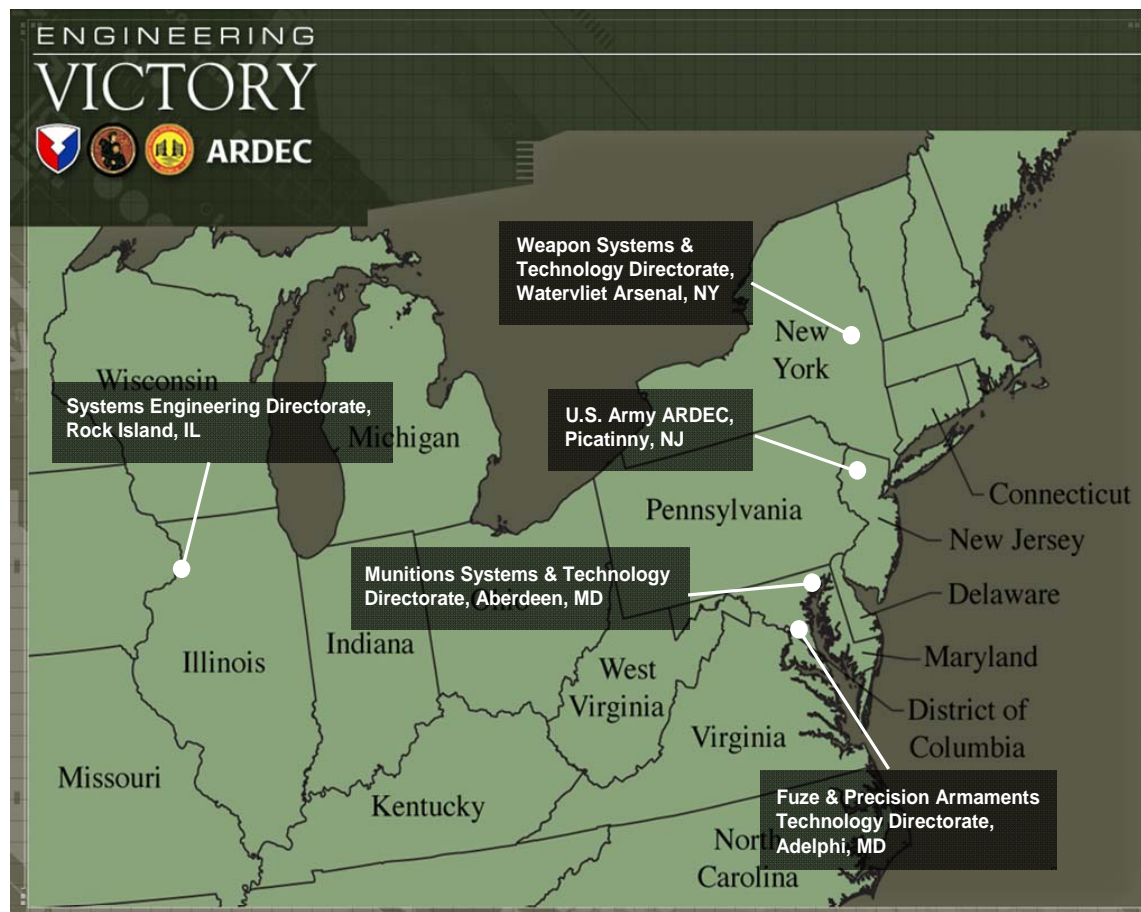
- Background Information
- Nanomaterials
- Picatinny's Production Capabilities
  - Bottom Up
  - Top Down
- Picatinny's Characterization Capabilities

*Powder technologies are an integral part of Picatinny's Mission*



## Picatinny Arsenal

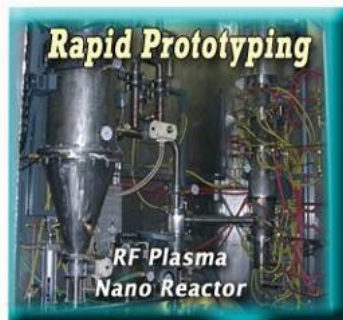
- “Home of American Firepower”
- Baldrige Award Winner
- Improving legacy items
- Developing new items
- Prototyping capability
- Fill technology gaps







# Nanotechnology Facility



## *Nanoparticle Reactor Facility at Picatinny Arsenal*

*Next generation materials for both military and commercial products*



**North America's largest RF Plasma facility for high performance nanomaterials**



An integrated state-of-the-art facility to synthesize, process, and characterize nanophase and nanostructured materials, fully dense near-net shape bulk components, and nanostructured coatings

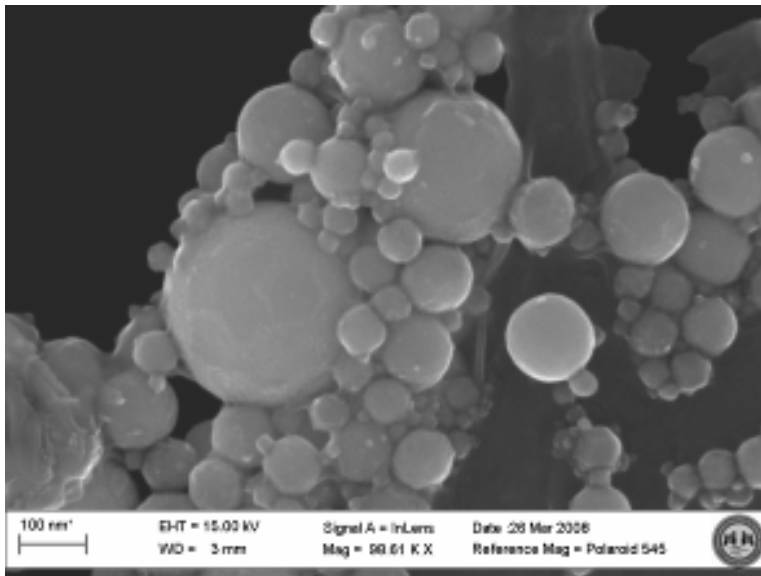


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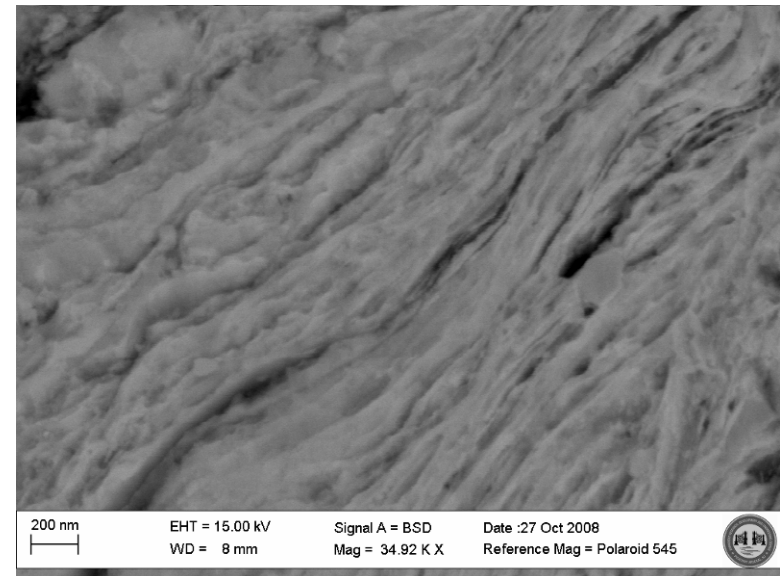
- Nanophase Materials

- Materials having nanoscale primary particle size



- Nanostructured Materials

- Materials which are not necessarily nanoscale but possess features which are on the nanoscale.





- Nanomaterial – a material having at least one dimension in the 1-100nm range
- Thickness of paper = 100,000nm
- Thickness of a human hair = 50,000nm
- Comparing a nanometer to a meter is like comparing a soccer ball to the earth
- There are 25,400,000 nanometers in 1 inch



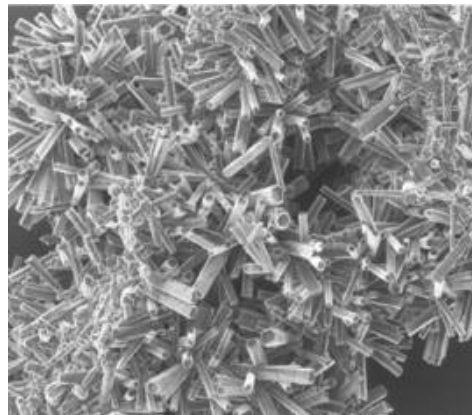
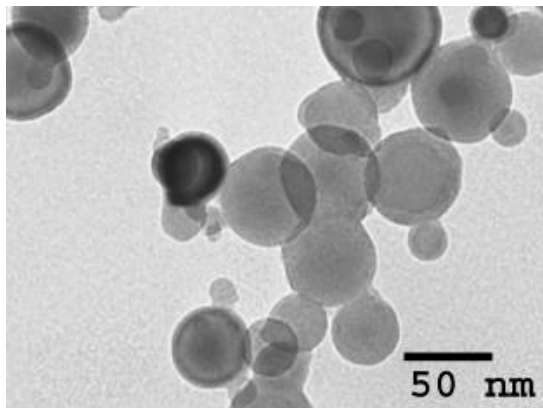
***Nanotechnology is a natural extension of particulate technology.***





- What happens on the nano scale

- Tunable properties → Particle morphology
- Higher strength
- Reduced Weight
- Increased reactivity → Increased surface area
- Increased durability



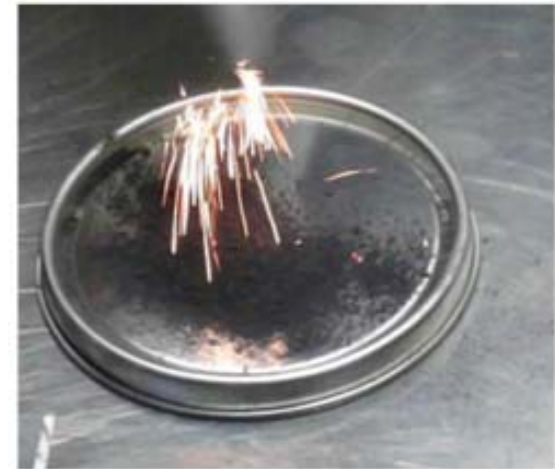
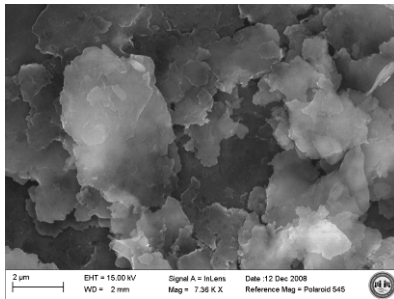




# Anti-Corrosion Applications



- Take conventional anti-corrosion techniques and scale down to the nano level
- Multi functionality as well as improved functionality
- Traditional properties may no longer apply between bulk and nanoscale
  - Materials become transparent
  - Materials become pyrophoric

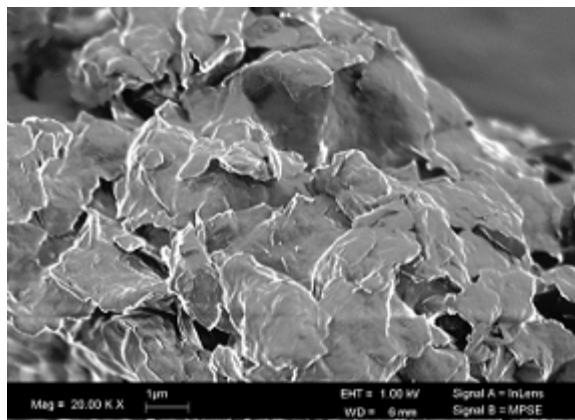
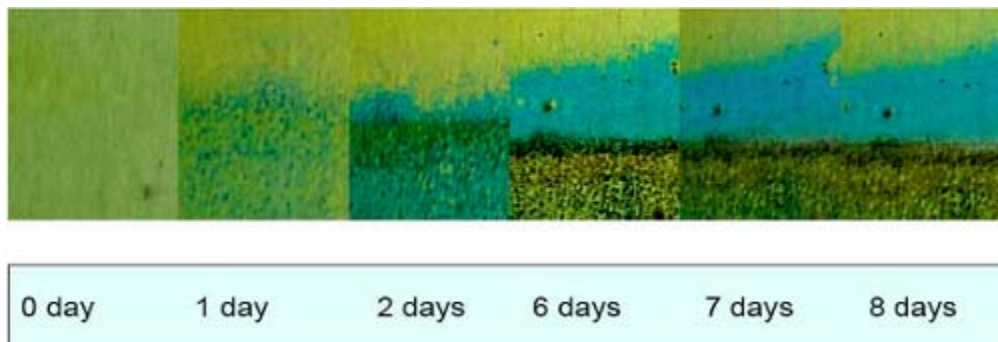


- **Surface modifications, protection, and functional coatings utilize powders and particulate technology**

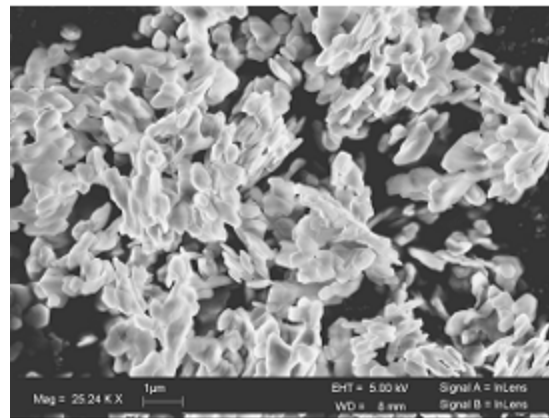




## Results after accelerated corrosion testing



**Montmorillonite (Cloisite 15A)  
/ 5% Bromothymol Blue**



**Hydrotalcite / 5%  
Thymophthalein**



- Bottom Up Approach



- Top Down Approach



*Using thermal plasma technology to synthesize nanomaterials is at the heart of our prototyping facility.*





# Inductively Coupled Plasma

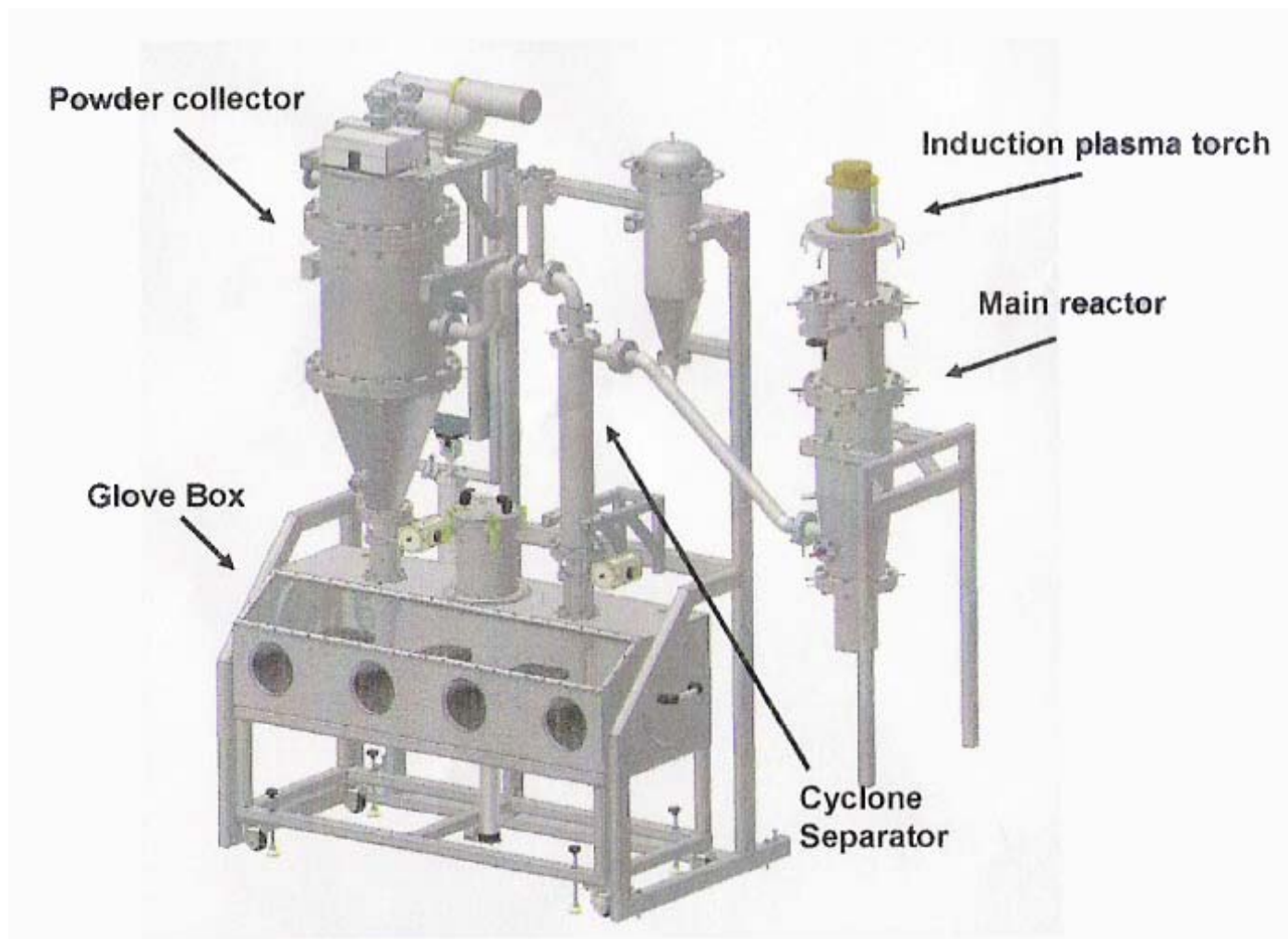


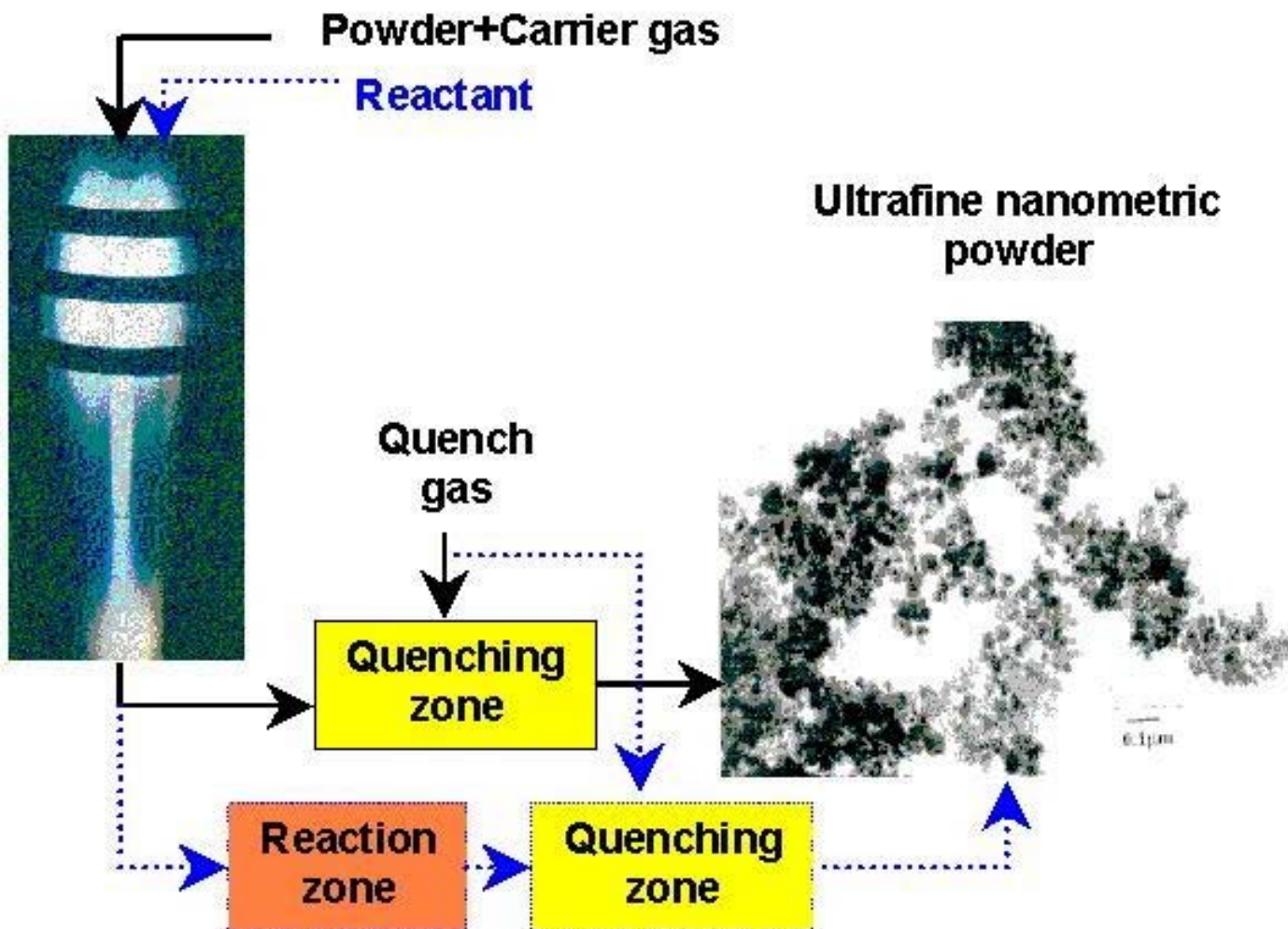
- Induction Plasma is a versatile and high rate technology to synthesize nano scale powders





# Reactor Layout

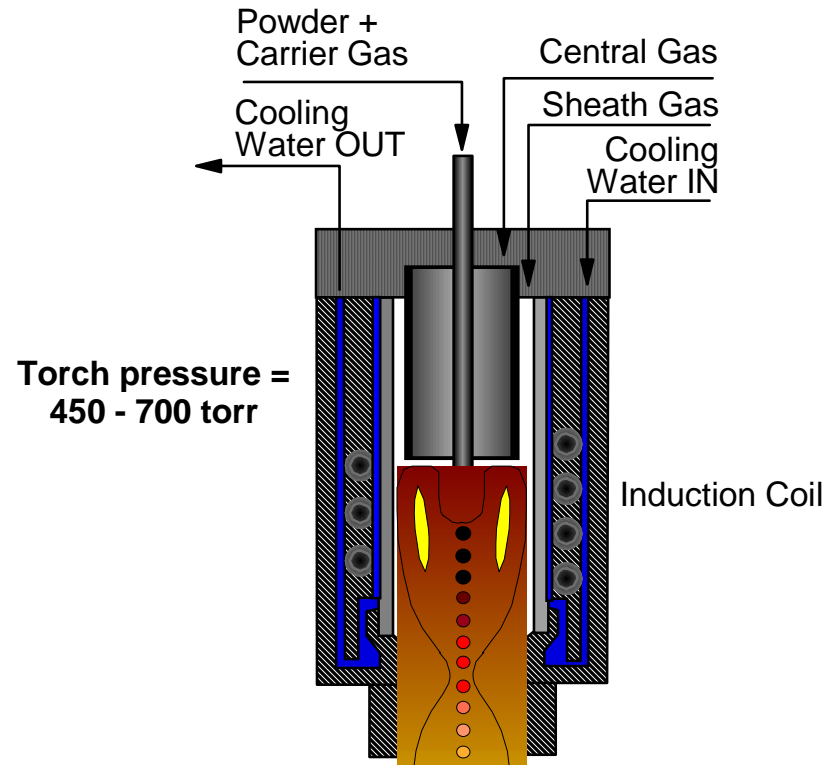






- Plasma Technology Benefits

- Powder or liquid feed stock
- No limit on material type
  - Over 10,000k plasma temperature
- High purity production
- Flexibility of operating conditions





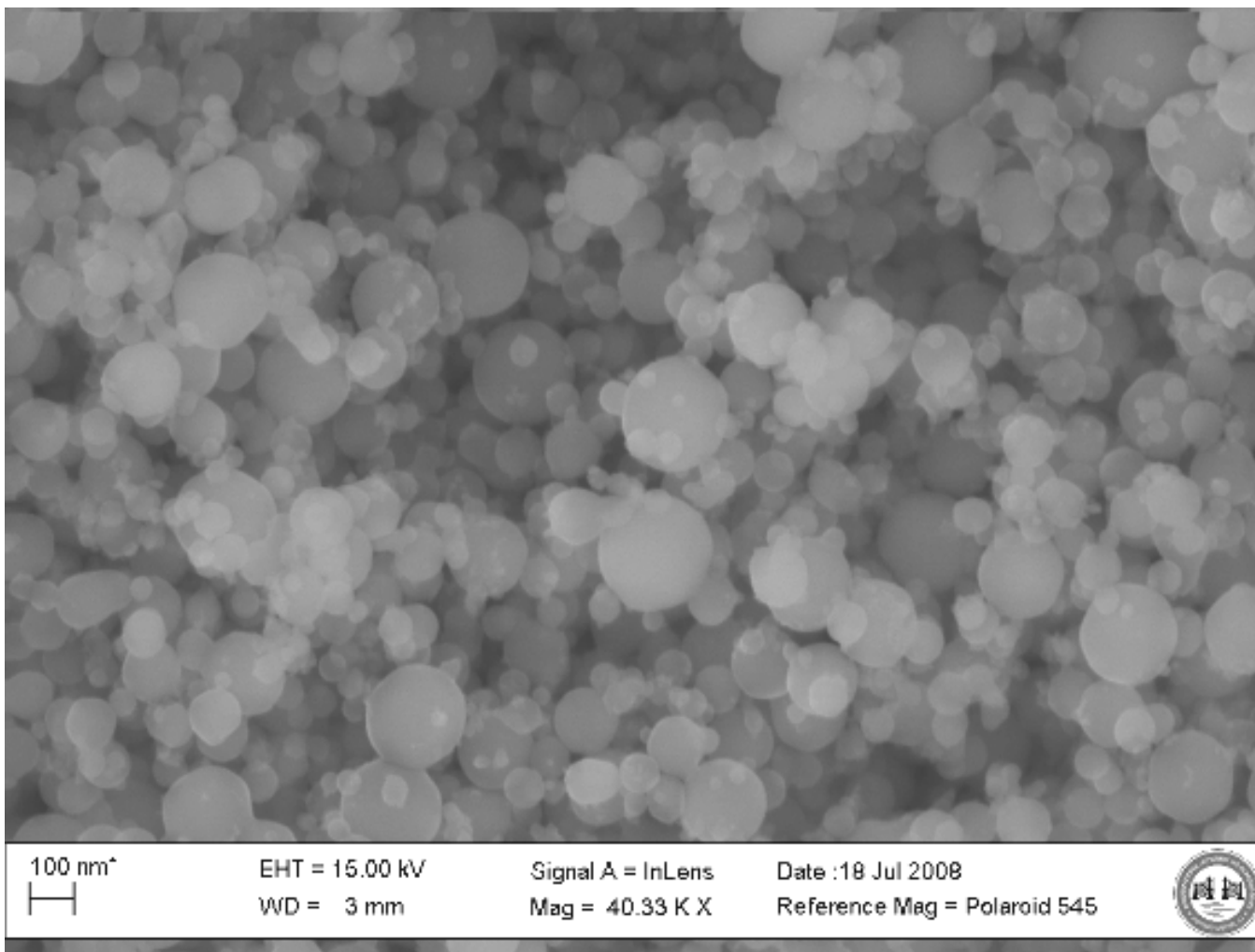


- Two Plasma Reactors
  - One unit for metals only
  - One unit for ceramics and non oxides
  - Production rate: up to 1kg/hr
- Some nanoscale powder examples
  - Aluminum
  - Tungsten
  - Cerium Oxide
  - Boron Carbide
  - Iron





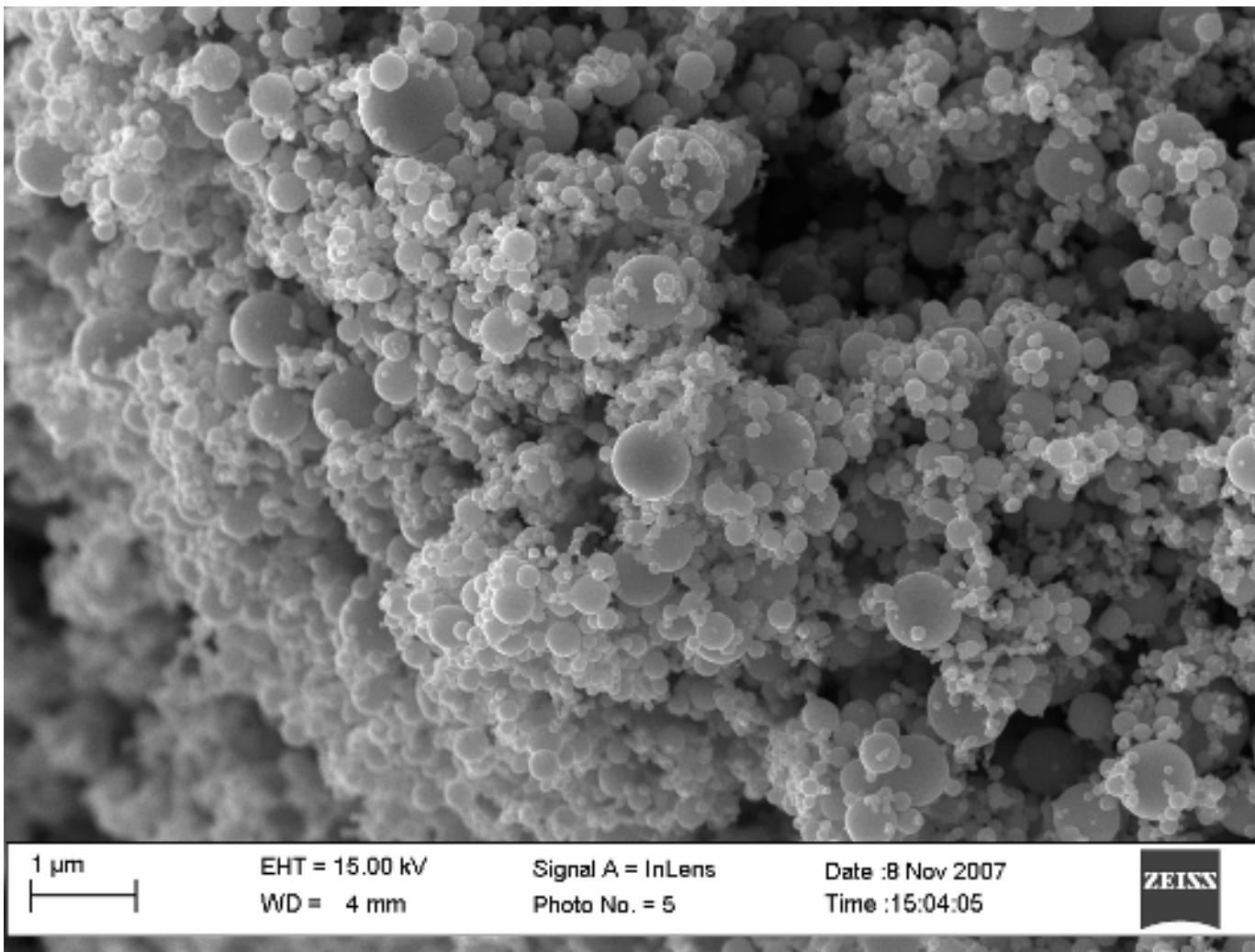
# Nano Aluminum



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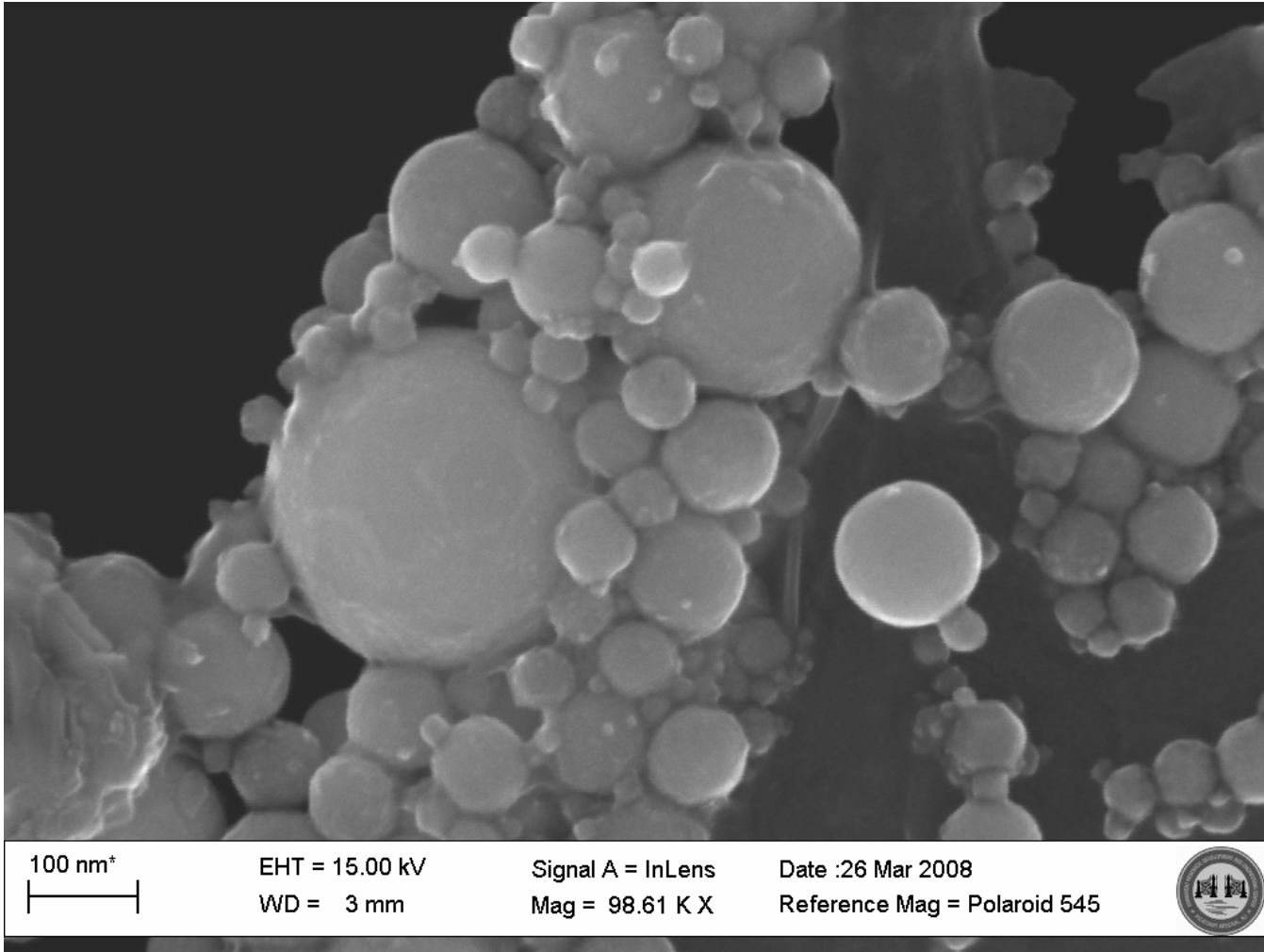


# Nano Iron

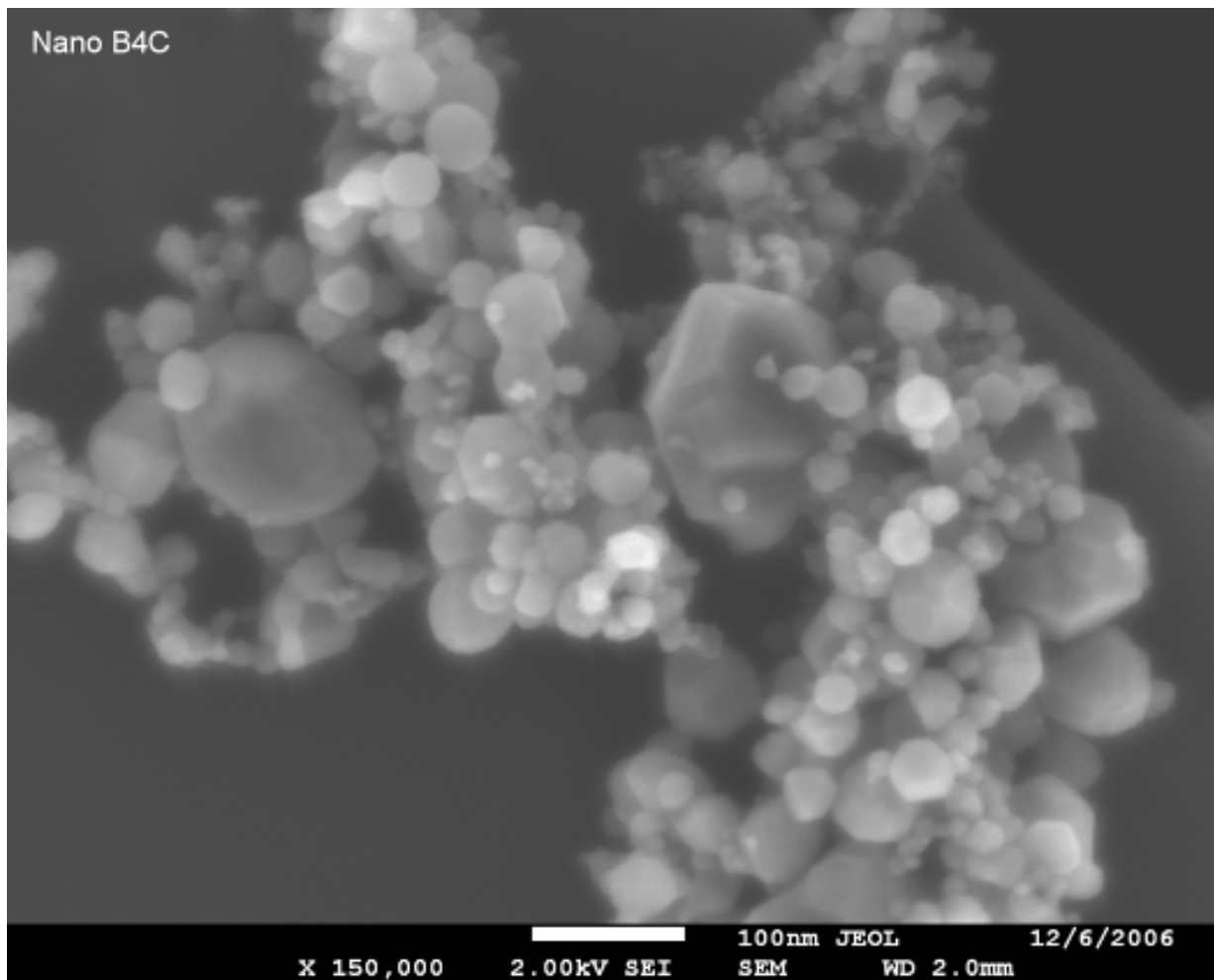




# Nano Tungsten









- High energy milling is a top down approach to fabricate nanostructured metals, alloys, ceramics, cermets, and reactive materials.



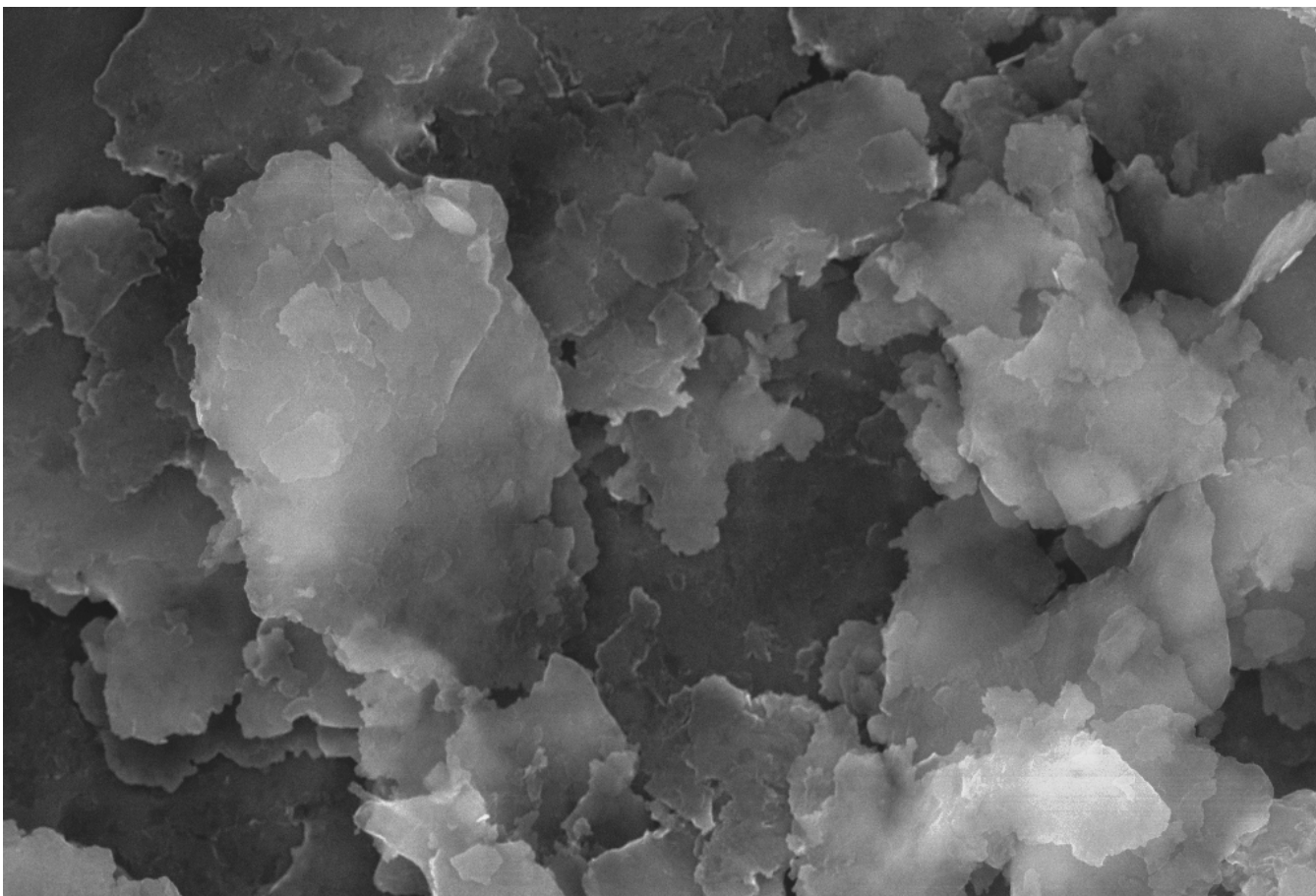
- Inert atmosphere for reactive materials
- Semi-continuous prototyping capability
- Experimental batch capability
- Tailor made compositions
- Three total units
  - Two (2) one liter machines
  - One (1) 8 liter machine



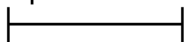
- **Able to impart nano sized grains into micron sized powders**



# Nanostructured Aluminum Flakes



2  $\mu$ m



EHT = 15.00 kV

WD = 2 mm

Signal A = InLens

Mag = 7.36 K X

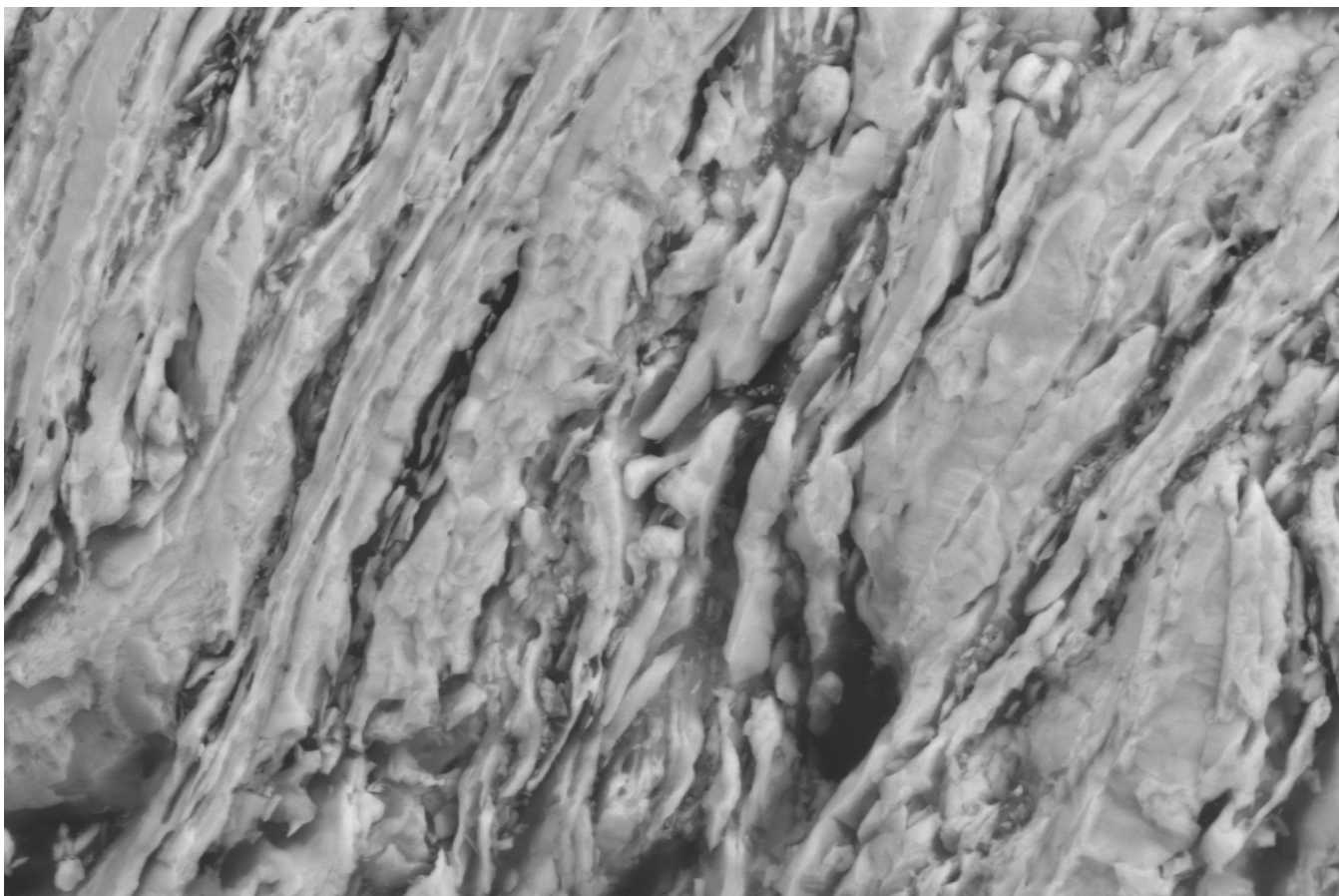
Date :12 Dec 2008

Reference Mag = Polaroid 545

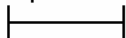


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1  $\mu\text{m}$



EHT = 15.00 kV

WD = 6 mm

Signal A = BSD

Mag = 9.74 K X

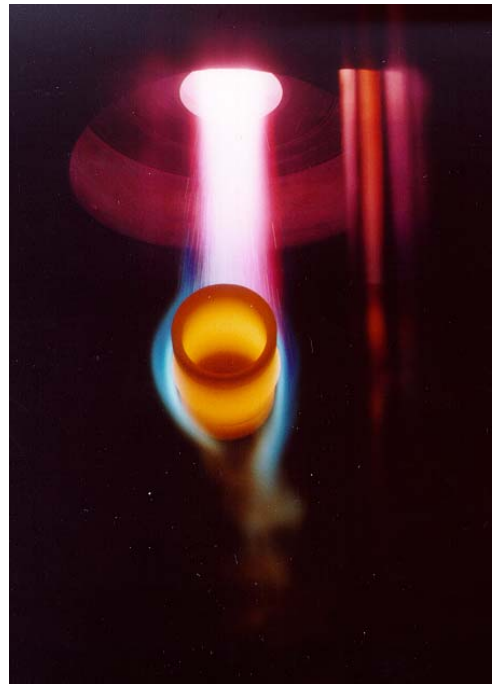
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Reference Mag = Polaroid 545





- Coatings can be applied via liquid or vapor deposition
- VPSD (Vacuum Plasma Spray Deposition)
- Can be used to net shape bulk products or apply coating layers





# Characterization Facilities

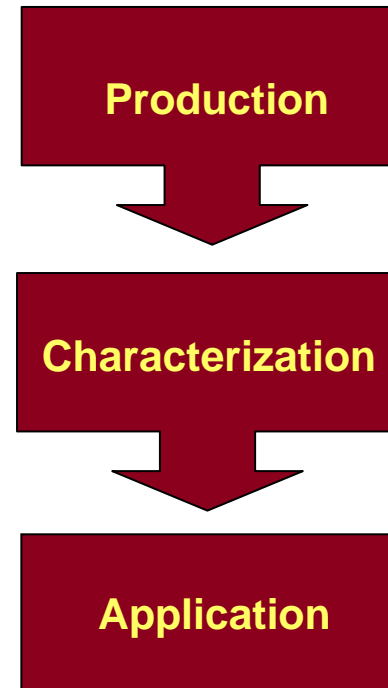


- Current equipment:
  - Field Emission Scanning Electron Microscope
  - X-ray Fluorescence
  - X-ray Diffractometer
  - Small angle X-ray scattering & Ultra small angle X-ray scattering
  - Thermal analysis equipment





- Particle size and distribution
- Composition
- Morphology
- Surface Area
- Thermal Properties
- Passivation layer thickness
- Crystallite size
- Phase identification



- Picatinny's facilities allow for rapid characterization of materials as soon as they are made.





- Zeiss Gemini Ultra Variable Pressure FE-SEM with EDX
  - Capable of low voltage
  - VP mode
  - STEM attachment
  - 1nm resolution @ 20kV
  - 12-1000000x magnification
  - High efficiency in-lens
  - Secondary and Backscatter
  - Used to determine:
    - Particle size
    - Distribution
    - Morphology
    - Passivation layer
    - Composition



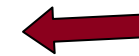


- Rigaku ZSX Primus II X-ray Fluorescence
  - Capable of holding up to 64 samples
  - Quantitative analysis results
  - Composition determination
  - Solid, liquid, or powder samples





**XRD Unit**



**SAXS**



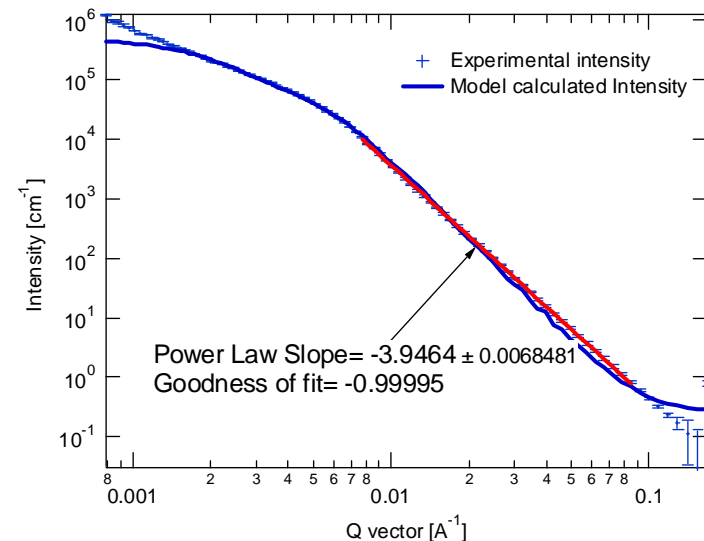
**USAXS**

- Rigaku Ultima XRD
  - Determines phase and crystallite size
  - Bulk sample holder
  - Hot stage
  - 6 ring powder sample holder





- Combining Small Angle and Ultra-small Angle X-ray Scattering allows *simultaneously* characterizing
  - Primary, secondary particle size & particle morphology
  - User friendly software developed
  - Total length scale, 1 nm – 2  $\mu\text{m}$





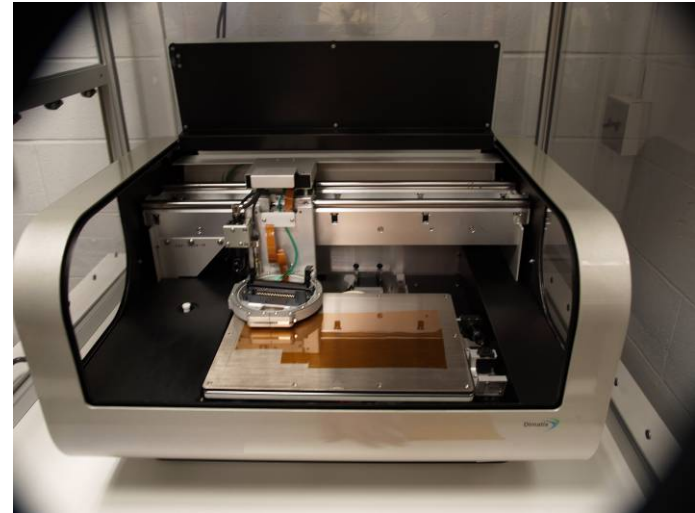


- Netzsch STA 449 C Jupiter – QMS 403 Aeolos Integrated TG-DSC / Mass Spec
  - Thermal properties
    - Oxidation
    - Exotherms
    - Endotherms
    - Melting Point
    - Moisture content
    - Organics content
  - Aging characteristics
  - Passivation layer thickness





- Nano ink printer
- Low voltage electron microscope
- Optical Microscopes
- Polishing and grinding units
- Spray dryer
- BET
  - Surface area
- Glow Discharge
  - Composition
- Oxygen analyzer





- **Powder Production Capability**

- Able to produce nanostructured materials with enhanced properties using high energy milling.

- Able to produce almost any nano scale powder at a rate up to 1kg/hr for potential applications involving anti-corrosion technologies.

- **Materials Characterization Capability**

- Collaboration also available for corrosion mechanism identification

- Able to obtain vast amounts of information about bulk materials or nanomaterials in a short time.



# THANK YOU



## Contact information

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